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# **Test Requirements for Coal-Tar Mixtures on Airport Pavements**

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Final Report

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The research documented in this report represents an effort to evaluate the test procedures for coal tar mixtures. The various coal tar test methods have been evaluated under several levels of test variables. The freeze-thaw and scuff resistance tests were evaluated under three types of shingles and an aluminum substrate. The fuel resistance test was evaluated under three levels of sand loading and three levels of film thickness. In addition, all of the tests were evaluated under two levels of humidity.

The analysis of the data indicates that the effect of the substrate is insignificant in all of the tests. The effect of humidity is significant on some tests, while the effects of sand loading and film thickness are highly significant on the results of the fuel resistance tests.

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#### INTRODUCTION

Coal tar emulsion sealers have historically been used to protect asphalt concrete pavements from oil, fuel, water, and weathering. Because sealers have an ability to resist freeze/thaw, they have been used extensively on airport taxiways, automobile parking areas, and fueling areas. Dripped oil can soften an asphalt concrete pavement. The sealers provide an impermeable surface to prevent fuel, oil and water intrusion which can lead to the raveling and/or stripping of the pavements. Coal tar sealers can also prevent weathering of an asphalt pavement by sealing it from sunlight and oxidation.

Sand is used with coal tar emulsions to enhance the skid resistance of the finished surface. Sand loading has been increased over recent years in an attempt to provide an increase in shape, but, this has resulted in problems with keeping the sand in suspension in the coal tar emulsions. Also, this sand/binder interface has provided a path for petroleum products to penetrate the sealer.

Experimental studies showed that the latex polymeric additives in coal tar emulsions could increase their ability to hold the sand in suspension. Another added benefit of the latex was an increase in the flexibility of the sealer. This flexibility allows the sealer to deflect with the underlying pavement as it contracts and expands with both thermal changes and traffic loads.

In 1988 a new set of specifications was developed for coal tar sealers based on an extensive laboratory testing program sponsored

by the Federal Aviation Administration (FAA) (1). The use of these specifications on several jobs has indicated that they suffer some limitations, perhaps because the original tests were conducted on a limited number of test variables. In the case of freeze/thaw and scuff resistance tests, the original program considered only one type of roofing shingle as a substrate and curing was accomplished under one level of humidity. Similarly, in the case of peel test, only one level of humidity was considered. In the fuel resistance test, the test variables were limited to one film thickness, one sand loading, and one humidity level.

Since coal tar emulsions are applied under various conditions of mix dcsign, existing pavement surface, and environmental conditions, the reliability range of the specifications is too narrow. In this study, the influence of the substrate, humidity, sand loading, and film thickness on the test results was evaluated. A laboratory experiment was designed to evaluate the reliability of the specifications when the conditions of the original tests are changed.

#### **OBJECTIVES**

The objectives of this study are as follows:

- To evaluate the effect of substrate on the results of the freeze/thaw and scuff tests.
- To evaluate the effect of humidity level on the results of the freeze/thaw, scuff, peel, and fuel resistance tests.

 To evaluate the effects of sand loading and film thickness on the results of the fuel resistance (tile) test.

In order to accomplish these objectives, an extensive laboratory testing program was conducted encompassing the various levels of test variables.

#### LABORATORY TESTING

Laboratory testing on coal tar emulsions is usually conducted in two phases. Phase 1 includes the selection of a mix design where the optimum water and additive contents are selected based on the testing sequence shown in Figure 1. Phase 2 is conducted after the mix design is selected, where quality control and quality assurance tests may be conducted on field samples to ensure the compliance of the product with the applicable specifications. The following represents a brief description of the test methods used in both phases.

All tests except the viscosity test use coal tar emulsion with sand; this is referred to as the composite system (coal tar, water, additive, and sand). The viscosity test is conducted on both the composite system and on the total liquid system (coal tar, water, and additive).

#### Viscosity Test

Viscosity is measured using the Brookfield viscometer DVII.

The limits for the viscosity are in the range of 10 - 90 poises,

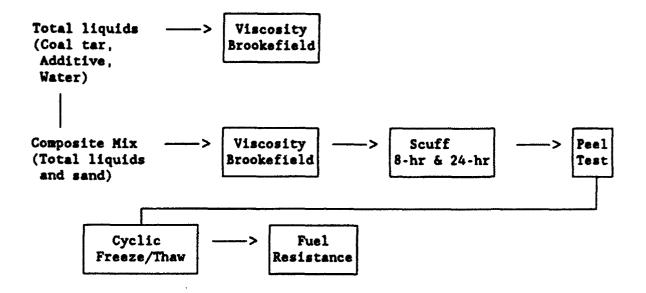


Fig. 1. Flow chart of the Mix Design

which represents the desirable limits established from previous research. These limits apply for both the total liquid system and the composite system. The total liquid system uses a combination of three levels of water and three levels of additive. Table 1 presents typical results from the laboratory test conducted in this research (1). The use of the viscosity test in this laboratory experiment was limited to the mix design phase only.

#### Scuff resistance

Scuff resistance is measured by the time required for the material to cure or set up. Measurement of "curetime" was developed by the slurry seal industry and modified during the previous research program at UNR (2). The test follows the ASTM D3910 procedure using a cohesion tester and measures both the rate of set and the final scuff resistance of the coal tar emulsion (3). This scuff resistance test was developed to determine when a newly sealed pavement could be opened to traffic, and also if the sealer provides adequate scuff resistance.

The test procedure consists of applying a uniform film thickness of coal tar emulsion to a substrate using a 16-gauge sheet metal mask. The mask is 6-inch by 6-inch with a 4-inch by 4-inch section removed from the center. A straight edge is used to apply the material within the mask. After curing for eight hours the sample is placed on the platen of the testing machine and held in place with "C" clamps. The platen is raised upward to the rubber abrasion head, and a normal stress of 73 psi is applied to

Table 1. Typical viscosity test results on both total liquid and composite mix.

Total Liquids: Check mix for incompatibility between coal tar emulsion and additive Viscosity Test, Limits: 10 - 90 poises.

|        |       | ADDITIVE |      |
|--------|-------|----------|------|
| WATER  | Low   | Medium   | High |
| Low    | 57.60 | 16.50    | X    |
| Medium | 25.40 | X        | X    |
| High   | 24.00 | X        | X    |

Composite Mix: Check Workability of Mix,

Viscosity Test, Limits: 10 - 90 poises.

|        |       | ADDITIVE |      |
|--------|-------|----------|------|
| WATER  | Low   | Medium   | High |
| Low    | 67.10 | 86.50    | x    |
| Medium | 27.80 | Х        | x    |
| High   | 21.30 | X        | X    |

Note: X indicates that the material fails to pass the specifications.

the sample through a calibrated proving ring. A torque wrench with a capacity of 300 inch-pound is then pulled through an arc of 180 degrees and the torque reading is taken in inch-pounds. This reading indicates the resistance of the rubber abrasion head on the sample. The same procedure is repeated on the cured sample at 24 hours. The 8-hour reading constitutes the initial set and the 24-hour reading gives the final scuff resistance. Based on the current specifications, limits are set at:

- 1. A minimum torque of 100 inch-pound @ 8 hours
- 2. A torque greater than the 8 hour reading at 24 hours
  Limits for scuff values at eight hours are set in order to provide
  a substantially scuff-resistant surface eight hours after placing
  materials. A minimum scuff value was established at 24 hours to
  indicate optimal scuff resistance for any given set of components.
  In this experiment the scuff resistance of the coal tar emulsions
  were evaluated using four different substrates and two levels of
  humidities during the curing period. The results are discussed in
  the data analysis section of this report.

#### Freeze-Thaw Test

The cyclic freeze-thaw conditioning test was developed from the Lottman accelerated procedure for predicting moisture-induced damage to asphalt concrete pavements (2). The procedure involves a series of freeze and thaw conditions designed to simulate thermal changes in a pavement in a northern climate. The test consists of applying coal tar emulsion on a 11-inch by 11-inch section

substrate using uniform film thickness of 1/16 inch. After initial curing, samples are placed in a 140°F oven for 24 hours, then moved to a 10°F for 24 hours. This procedure constitutes one freeze thaw cycle. Samples are monitored for cracking after five and ten cycles, using a commercially available thickness gauge and grid frame which was developed in the previous research program for rating the severity of the cracking (2). The current specifications call for the following limits:

- 1. 1 or less at the end of 5 cycles.
- 2. 3 or less at the end of 10 cycles.

The severity of cracking is rated as follows:

| Rating | Severity of cracking | Width of widest crack | Percent of cracking          |
|--------|----------------------|-----------------------|------------------------------|
| 1      | Hairline cracking    | 0.010mm               | NA-Cracks are barely visible |
| 2      | Slight cracking      | 0.015mm               | <25%                         |
| 3      | Moderate cracking    | 0.020mm               | >25%                         |
| 4      | Severe cracking      | 0.020mm or greater    | >50%                         |

In this experiment the freeze thaw resistance of the coal tar emulsions was evaluated using four different substrates and two levels of humidity during the curing period. The results are discussed in the data analysis section of this report.

#### Adhesion or Peel Test

The objective of this test is to predict the loss of adhesion

between the coal tar sealer and the pavement. The test for adhesion is based on ASTM D3359 'Measuring Adhesion by Tape Test' Method A, X-cut tape test (4).

A coal tar emulsion mixture is applied to two 3-inch by 6-inch aluminum panels using a 16-gauge sheet metal mask. The mask is 3-inch by 6-inch with a 2-inch by 4-inch section removed from the center. After application of material, the sample is allowed to cure for 24 hours at approximately 77°F and a selected level of humidity. After the completion of curing, an 'X' is cut into the sealer using a sharp knife for the panel to be visible. The 'X' is then covered with a pressure sensitive tape (40 oz/inch width). The tape is peeled back after 45 seconds and the adhesion between the sealer and the panel is measured.

The scale used to measure adhesion is defined by ASTM D3359 and is as follows:

- 5A No peeling or removal.
- 4A Trace peeling or removal along incisions.
- 3A Jagged removal along most of incision up to 1/16 inch on either side.
- 2A Jagged removal along most of incision up to 1/8 inch on either side.
- 1A Removal from most of the area of the 'X' under tape.
- OA Removal beyond the area of the 'X'.

The 'A' designation after the numerical rating indicates that Method 'A' is used in the testing. A rating of 5A is needed. In this experiment, the peel test was used to evaluate the adhesion of

coal tar emulsions under two levels of humidity. The results are discussed in the data analysis section of this report.

#### Fuel Resistance (Tile) Test

A significant amount of damage occurs to asphalt concrete pavements each year due to spillage of fuel, oil and hydraulic fluids. One method of reducing the damage is to seal the pavement with a coal tar emulsion seal coat. The test method comes directly from ASTM D3320 'Emulsified coal tar pitch (Mineral colloid type)' (5).

A film of coal tar emulsion mixture is applied to two 6-inch by 6-inch white unglazed ceramic tile. A uniform thickness is applied using a 16 gauge sheet metal mask. The mask is 6-inch by 6-inch with a 4-inch by 4-inch section removed from the center. The sample is then allowed to cure for 96 hours at 77°F and at a specified level of humidity. After the curing stage, a brass ring (2-inches in diameter and 2-inches high) is affixed to the sealer with silicon rubber, then the brass ring is filled with kerosene. After 24 hours, the coating is evaluated for the loss of fuel through penetration into the sealer.

The results are measured on a pass/fail basis. Visible evidence of leakage or discoloration of the tile after the tile is broken into half to expose the part of the tile that was subjected to the kerosene constitutes failure. In this experiment, the tile test was used to evaluate the fuel resistance of coal tar emulsions using three levels of film thickness, three levels of sand loading,

and two levels of humidity. The results are discussed in the data analysis section of this report.

#### **MATERIALS**

A total of four sources of coal tar emulsions were tested in this program. The physical properties of the coal tar emulsions and additives are summarized in Tables 2 and 3. Table 4 summarizes the levels of water, additive, and sand used in the formulation of the materials.

#### **DATA ANALYSIS**

As mentioned earlier, the objectives of this experiment were to evaluate the variability of test methods as a function of the levels of test variables. The laboratory tests summarized in Tables 5 & 6 were conducted. The mix design used in each test was selected based on two options:

- a) The mix design conducted in the University's laboratory,
- b) The mix design recommended by the manufacturer.

A total of four sources of coal tar emulsions were tested. It was anticipated that the four sources, the two types of mix designs, the two levels of humidity, the four types of substrates, and the three levels of sand loadings would generate a large data base, based on which significant recommendations could be made. The summary of the mix designs conducted in the University laboratory is presented in appendix A.

Table 2. Physical Properties of Coal Tar Emulsions.

|                                  |      | sol        | URCE |                 |
|----------------------------------|------|------------|------|-----------------|
| PROPERTY                         |      | <b>. 2</b> | 3    | (1917) <b>4</b> |
| Solids, %<br>(ASTM D2939)        | 49.1 | 51.0       | 50.0 | NA              |
| Specific Gravity<br>(ASTM D2939) | 1.21 | 1.20       | 1.20 | NA              |

Table 3. Physical Properties of Additives.

|                  |       | SOURCE |        |       |  |  |  |  |
|------------------|-------|--------|--------|-------|--|--|--|--|
| PROPERTY         |       | 2      | 3      | 4     |  |  |  |  |
| Solids, %        | 40    | 40     | NA     | NA    |  |  |  |  |
| Color            | Black | White  | Green  | Black |  |  |  |  |
| Specific Gravity | 1.003 | NA     | 1.02   | NA    |  |  |  |  |
| Silicon, %       | 0.0   | NA     | < 1.00 | NA    |  |  |  |  |

Table 4. Variable Levels used in the Experiment.

| VARIABLE | CODE   | QUANTITY                           |
|----------|--------|------------------------------------|
|          | Low    | 4.0 gal/100 gal coal tar emulsion  |
| ADDITIVE | Medium | 14.5 gal/100 gal coal tar emulsion |
|          | High   | 25.0 gal/100 gal coal tar emulsion |
|          | Low    | 20.0 gal/100 gal coal tar emulsion |
| WATER    | Medium | 55.0 gal/100 gal coal tar emulsion |
|          | High   | 90.0 gal/100 gal coal tar emulsion |
|          | Low    | 2.0 lb/gal coal tar emulsion       |
| SAND     | Medium | 7.5 lb/gal coal tar emulsion       |
|          | High   | 13.0 lb/gal coal tar emulsion      |

Table 5: Suggested Test Matrix for Evaluating the Effects of Substrate and Humidity.

| Coal Tar |                            |               | 13-20% Relative<br>Humidity | telative<br>lity |                |               | 40-80% Relative<br>Humidity | elative<br>lity |                |
|----------|----------------------------|---------------|-----------------------------|------------------|----------------|---------------|-----------------------------|-----------------|----------------|
| Source   | Additive                   | Shingle<br>#1 | #2                          | £#               | Alum.<br>3/16" | Shingle<br>#1 | 7.#                         | £#              | Alum.<br>3/16" |
|          | Mix<br>Design              | X             | X                           | X                | Y              | X             | X                           | X               | Y              |
|          | Manufacturer<br>Recomended | Х             | ×                           | ×                | Y              | ×             | ×                           | ×               | Y              |
|          | Mix<br>Design              | X             | ×                           | ×                | Y              | X             | ×                           | ×               | Y              |
| 2        | Manufacturer<br>Recomended | X             | ×                           | X                | Y              | X             | ×                           | ×               | Y              |
|          | Mix<br>Design              | ×             | Х                           | Х                | Y              | ×             | X                           | X               | Å              |
| က        | Manufacturer<br>Recomended | ×             | ×                           | X                | Y              | Х             | ×                           | X               | Ÿ              |
|          | Mix<br>Design              | ×             | ×                           | ×                | <b>&gt;</b>    | X             | ×                           | X               | Y              |
| 4        | Manufacturer<br>Recomended | ×             | ×                           | ×                | Y              | ×             | ×                           | ×               | Y              |

X = Freeze/Thaw, and Scuff Testing. Y = Freeze/Thaw, and Scuff, and Peel Testing.

Table 6: Test Matrix for Evaluating Effects of Film Thickness, Sand Loadings and Humidity on the Fuel Resistance of Coal Tar.

| Additive     | Film Thickness | Sand Loading | Humidity<br>13-20% | Humidity<br>40-80% |
|--------------|----------------|--------------|--------------------|--------------------|
|              |                | Low          | х                  | x                  |
|              | 1/16"          | Medium       | Х                  | Х                  |
|              |                | High         | х                  | х                  |
|              |                | Low          | х                  | x                  |
| Mix Design   | 1/8"           | Medium       | х                  | X                  |
| M            |                | Hìgh         | Х                  | X                  |
|              |                | Low          | Х                  | x                  |
|              | Manufacturer   | Medium       | Х                  | x                  |
|              |                | High         | Х                  | х                  |
|              |                | Low          | х                  | Х                  |
|              | 1/16"          | Medium       | Х                  | X                  |
|              |                | High         | х                  | X                  |
| Manufacturer | 4.404          | Low          | X                  | х                  |
|              | 1/8"           | Medium       | х                  | х                  |
|              |                | High         | х                  | X                  |
|              |                | Low          | х                  | X                  |
|              | Manufacturer   | Medium       | х                  | X                  |
|              |                | High         | Х                  | X                  |

X = Tile Testing

#### Analysis of the Scuff Resistance Data

The objective of this part of the research was to evaluate the effect of substrate and the effect of humidity on the scuff resistance of coal tar sealers used on asphalt concrete pavements.

The effect of the substrate was evaluated through testing three shingles from three different manufacturing companies and a 1/16-inch aluminum panel. The effect of humidity was evaluated by curing the coal tar emulsion mixture after application on the substrates under two different humidities of 15 percent and 60 percent in a specially constructed temperature and humidity controlled room.

#### Effect of the Substrate on Scuff Resistance

The results of the scuff resistance tests are summarized in Tables 7 and 8. The specification limits on the scuff resistance test require a minimum of 100 inch-pound torque after 8-hours and a higher torque at 24-hours. It should be noted that the final decision from the scuff resistance test is whether to accept or reject a coal tar emulsion based on the level of torque.

The data in Table 7, show that changing the type of substrate influenced the decision to pass or fail a coal tar emulsion (refer to test limits) only in the following cases:

Source 1, Manufacturer's recommended mix design.

Source 1, Laboratory recommended mix design.

Source 2, Manufacturer's recommended mix design.

The data in Table 5, which presents similar data except under

Table 7. Scuff Resistance Data, Low Humidity (inch-pounds)

| SOURCE MIV |                | Alumi<br>panel | inium        | Shing | gle #1 | Shing | ele #2 | Shing | le #3 |  |
|------------|----------------|----------------|--------------|-------|--------|-------|--------|-------|-------|--|
| SOURCE     | MIX-<br>DESIGN |                | Curing hours |       |        |       |        |       |       |  |
|            |                | 8              | 24           | 8     | 24     | 8     | 24     | 8     | 24    |  |
| _          | Manuf          | 75             | 180          | 85    | 160    | 105   | 185    | 110   | 160   |  |
| l          | Lab            | 40             | 150          | 140   | 150    | 150   | 135    | 125   | 165   |  |
|            | Manuf          | 95             | 170          | 90    | 170    | 110   | 190    | 120   | 210   |  |
| 2          | Lab            | 165            | 210          | 170   | 220    | 175   | 200    | 150   | 190   |  |
|            | Manuf          | 70             | 165          | 75    | 160    | 80    | 170    | 60    | 170   |  |
| 3          | Lab            | 110            | 130          | 140   | 160    | 140   | 165    | 100   | 125   |  |
|            | Manuf          | 160            | 170          | 190   | 205    | 210   | 235    | 200   | 225   |  |
| 4          | Lab            | х              | х            | Х     | Х      | Х     | х      | Х     | X     |  |

Table 8. Scuff Resistance Data, High Humidity (inch-pounds)

|        |                | Alumi<br>panel | nium | Shing | gle #1 | Shing | le #2 | Shing | le #3 |
|--------|----------------|----------------|------|-------|--------|-------|-------|-------|-------|
| SOURCE | MIX-<br>DESIGN | Curing Hours   |      |       |        |       |       |       |       |
|        |                | 8              | 24   | 8     | 24     | 8     | 24    | 8     | 24    |
|        | Manuf          | 90             | 130  | 95    | 100    | 90    | 150   | 95    | 120   |
| 1      | Lab            | 45             | 185  | 125   | 165    | 100   | 175   | 175   | 185   |
|        | Manuf          | 30             | 130  | 75    | 210    | 55    | 140   | 50    | 165   |
| 2      | Lab            | 135            | 160  | 160   | 220    | 155   | 210   | 150   | 195   |
|        | Manuf          | 70             | 165  | 60    | 145    | 80    | 180   | 55    | 160   |
| 3      | Lab            | 70             | 130  | 75    | 150    | 75    | 150   | 90    | 120   |
|        | Manuf          |                | 175  | 135   | 190    | 165   | 250   | 130   | 220   |
| 4      | Lab            | Х              | Х    | Х     | X      | X     | X     | Х     | X     |

high humidity (60%), show that the decision was influenced in only one case:

Source 1, Manufacturer's recommended mix design.

Based on the above observations, it can be recommended that in general the results of the scuff resistance test are not influenced by the type of substrate used.

#### Effect of Humidity on Scuff Resistance

The next step in this analysis was to evaluate the effect of humidity on the outcome of the scuff resistance test. In order to conduct this evaluation, it was necessary to compare the data elements in Tables 7 and 8 while recognizing that the minimum required torque should be 100 inch-pound. In other words, the effect of humidity was not considered significant unless it changed the torque to a level where the decision to accept or reject a product was influenced. For example, by changing the level of humidity, the torque changes from 50 to 75 inch-pounds. Even though the absolute change in torque is large, it would not influence the decision. Therefore, the effect of humidity level in this case would be considered insignificant.

Using the above guidelines in evaluating the data in tables 7 and 8, it can be concluded that the humidity level is significant in the following cases:

Source 1, Manufacturer's recommended mix design

Source 2, Manufacturer's recommended mix design

Source 3, Laboratory's recommended mix design

The humidity level is significant for all four types of substrates in the above cases. Therefore, it can be concluded that the humidity level has a significant effect on the results of the scuff resistance test.

#### Analysis of the Adhesion or Peel Test Data

The objective of this part of the experiment was to evaluate the effect of humidity on the adhesion of coal tar sealers used on asphalt concrete pavements. The effect of the humidity was evaluated through testing coal tar emulsions cured in two different relative humidities of 15 percent and 60 percent in temperature and humidity controlled rooms for 24 hours. The results of this test are summarized in Table 9. Based on this data, it can be observed that the effect of humidity on the adhesion test for the coal tar emulsions is insignificant.

#### Analysis of Freeze-Thaw Cracking Data

The objective of this part of the research was to study the effect of substrate and the effect of humidity on the freeze-thaw resistance of the coal tar emulsions used on asphalt concrete pavements.

The effect of substrate was evaluated through testing three types of shingles and a 3/16-inch aluminum panel. The shingles were manufactured by three different companies from western states. The shingles were cut to 12 by 12 inches square and coal tar composite mixtures were applied, using the mask on all the four

Table 9. Adhesion or Feel Test Rating Data

| Source                                 | Mix Design   | Low Humidity | High Humidity |
|--|--------------|--------------|---------------|
|  | Manufacturer | 5A           | 5A            |
| 1000 1000 1000 1000 1000 1000 1000 100 | Laboratory   | 5A           | 5A            |
|  | Manufacturer | 4A           | 4A            |
| 2                                      | Laboratory   | 5A           | 5A            |
|  | Manufacturer | 5A           | 5A            |
| 3                                      | Laboratory   | 5A           | 5A            |
| 4                                      | Manufaturer  | 5A           | 5A            |
|  | Laboratory   | Х            | x             |

substrates. After application, the samples were allowed to cure at 77° F under two different humidity levels of 15 percent and 60 percent for 24 hours in a temperature and humidity controlled room. After five and ten freeze-thaw cycles the samples were monitored for cracking using the crack measuring grid and feeler gauges, and they were rated from 1 to 4 on the rating scale as described in the test procedure. The results of the freeze-thaw tests are summarized in Tables 10 and 11.

#### Effect of Substrate on Freeze-Thaw Data

The specification limits of the freeze-thaw test call for the following:

- 1. Acceptable rating of 1 after 5 cycles
- 2. Acceptable rating of 3 after 10 cycles

The evaluation of the freeze-thaw data follows the same approach used in the evaluation of the scuff resistance data. The effect of any variable is considered significant if a change in its level causes a change in the final recommendation of the test. Table 10 summarizes the test data under the low humidity level. A change in the type of the substrate changed the decision to accept or reject a material only in the case of source 1, manufacturer's recommended mix design. Therefore it can be concluded that the effect of substrate under the low humidity level is insignificant.

Table 11 summarizes the test data under the high humidity level. Changing the type of substrate changes the decision to accept or reject a material only in the case of source 1,

Table 10. Freeze-Thaw Rating Data after 5 and 10 Cycles, Low Humidity

|        |                |   | inium | Shing | gle #1 | Shing  | ;le #2 | Shing | le #3 |
|--------|----------------|---|-------|-------|--------|--------|--------|-------|-------|
| SOURCE | MIX-<br>DESIGN |   |       |       | No of  | Cycles |        |       |       |
|        |                | 5 | 10    | 5     | 10     | 5      | 10     | 5     | 10    |
|        | Manuf          | 1 | 1     | 2     | 4      | 4      | 4      | 1     | 3     |
| 1      | Lab            | 1 | 1     | 1     | 2      | 1      | 3      | 1     | 3     |
|        | Manuf          | 1 | 1     | 1     | 1      | 1      | 1      | 1     | 1     |
| 2      | Lab            | 1 | 1     | 1     | 1      | 1      | 1      | 1     | 1     |
|        | Manuf          | 1 | 1     | 1     | 2      | 1      | 3      | 1     | 1     |
| 3      | Lab            | 1 | 1     | 1     | 1      | 1      | 1      | 1     | 1     |
| Manuf  |                | 4 | 4     | 4     | 4      | 4      | 4      | 4     | 4     |
| 4      | Lab            | Х | Х     | Х     | X      | X      | X      | Х     | Х     |

Table 11. Freeze-Thaw Rating Data after 5 and 10 Cycles, High Humidity

|        |                | Alum<br>panel | inum | Shing | gle #1 | Shing  | le #2 | Shing | le #3 |
|--------|----------------|---------------|------|-------|--------|--------|-------|-------|-------|
| SOURCE | MIX-<br>DESIGN |               |      |       | No of  | Cycles |       |       |       |
|        |                | 5             | 10   | 5     | 10     | 5      | 10    | 5     | 10    |
|        | Малиf          | 1             | 1    | 1     | 3      | 1      | 3     | 3     | 4     |
| 1      | Lab            | 1             | 1    | 2     | 3      | 1      | 1     | 3     | 4     |
| _      | Manuf          | 1             | 1    | 1     | 3      | 1      | 1     | 1     | 4     |
| 2      | Lab            | 1             | 1    | 1     | 1      | 1      | 2     | 1     | 1     |
| _      | Manuf          | 1             | 1    | 1     | 2      | 1      | 3     | 1     | 1     |
| 3      | Lab            | 1             | 1    | 1     | 1      | 1      | 1     | 1     | 1     |
|        | Manuf          | 4             | 4    | 4     | 4      | 4      | 4     | 4     | 4     |
| 4      | Lab            | Х             | Х    | х     | X      | х      | x     | Х     | х     |

manufacturer's and laboratory's recommended mix design. Since only one source shows significant effect from the substrate, it can be concluded that, overall, the effect of substrate under high humidity is insignificant.

#### Effect of Humidity Level on Preeze-Thaw Cracking

In order to evaluate the effect of humidity level on the freeze-thaw cracking of coal tar emulsions, the data in Tables 10 and 11 were individually cross-checked. Each set of data was compared at both levels of humidity. If there was a high number of cases in which the decision changed as a result of changing the humidity level, then the effect of humidity level was significant. A change in the level of humidity caused a change in the decision to accept or reject a material in the following cases:

Source 1, Manufacturer's recommended mix design, Shingle # 1
Source 1, Manufacturer's recommended mix design, Shingle # 2
Source 1, Manufacturer's recommended mix design, Shingle # 3

Source 1, Laboratory's recommended mix design, Shingle # 3

Source 2, Manufacturer's recommended mix design, Shingle # 3 In general, the level of humidity was significant on one out of four sources of coal tar emulsion. The level of humidity was significant only in five cases. Therefore, it can be concluded that the humidity level has an insignificant effect on the results and recommendations of the freeze-thaw cracking test.

#### Analysis of Fuel Resistance (Tile) Test Data

The objective of this part of the experiment was to evaluate the effect of film thickness, sand loading, and humidity level on the results of the fuel resistance test of coal tar emulsions. Tables 12 through 15 summarize the fuel resistance data for all four sources of coal tar emulsions. Again, in order to evaluate the effect of a variable on the test results, it was necessary to investigate its significance on the final recommendation of the test. The sand loading levels used in this experiment included low (2 pounds/gallon), medium (7.5 pounds/gallon), and high (13 pounds/gallon)

#### Effect of Film Thickness on Fuel Resistance

The data in Tables 12 through 15 show that the results of the test were affected when a 1/16 inch film thickness was used as compared to the 1/8 inch and manufacturer's recommended film thickness. This discrepancy between the 1/16 inch film and other thicknesses appears in almost all cases. In the majority of the cases, the manufacturer's recommended film thickness is either 1/8 inch or multiple films of 1/16 inch. Therefore it may be concluded that film thickness has a significant effect on the result. However, as long as the final film thickness is 1/8 inch, regardless of the method of application (one 1/8 inch or two 1/16 inch films), the results would be consistent.

Table 12. Fuel Resistance Data (Tile Test), Source # 1

| Additive     | Film Thickness                         | Sand Loading | Humidity<br>13-20% | Humidity<br>40-80% |      |
|--------------|--|--------------|--------------------|--------------------|------|
|              |  | Low          | Pass               | Fail               |      |
|              | 1/16"                                  | Medium       | Fail               | Fail               |      |
|              |  | High         | Fail               | Fail               |      |
|              |  | Low          | Pass               | Pass               |      |
| Mix Design   | 1/8"                                   | Medium       | Pass               | Pass               |      |
|              |  | High         | Fail               | Fail               |      |
|              |  | Low          | Pass               | Pass               |      |
|              | Manufacturer                           | Medium       | Pass               | Pass               |      |
|              |  | High         | Fail               | Fail               |      |
| ·            |  | Low          | Fail               | Pass               |      |
|              | 1/16"                                  | Medium       | Fail               | Fail               |      |
|              |  | High         | Fail               | Fail               |      |
|              | _                                      | Low          | Pass               | Pass               |      |
| Manufacturer | 1/8"                                   | Medium       | Pass               | Pass               |      |
|              |  | High         | Fail               | Fail               |      |
|              | •••••••••••••••••••••••••••••••••••••• |              |                    | Pass               | Pass |
|              | Manufacturer                           | Medium       | Fail               | Pass               |      |
| ·            |  | High         | Fail               | Fail               |      |

Table 13. Fuel Resistance Data (Tile Test), Source # 2

| Additive          | Film Thickness    | Sand Loading | Humidity<br>13-20% | Humidity<br>40-80% |
|-------------------|-------------------|--------------|--------------------|--------------------|
|                   |                   |              | Fail               | Fail               |
|                   | 1/16"             | Medium       | Fail               | Fail               |
|                   |                   | High         | Fail               | Fail               |
|                   |                   | Low          | Pass               | Pass               |
| Mix Design        | 1/8"              | Medium       | Pass               | Pass               |
|                   | ·                 | High         | Fail               | Fail               |
|                   |                   | Low          | Pass               | Pass               |
|                   | Manufact-<br>urer | Medium       | Pass               | Pass               |
|                   |                   | Medium I     | Fail               | Fail               |
|                   |                   | Low          | Fail               | Fail               |
|                   | 1/16"             | Medium       | Fail               | Fail               |
|                   |                   | High         | Fail               | Fail               |
|                   |                   | Low          | Pass               | Pass               |
| Manufact-<br>urer | 1/8"              | Medium       | Fail               | Fail               |
| ų i e i           |                   | High         | Fail               | Fail               |
|                   | _                 | Low          | Pass               | Pass               |
|                   | Manufact-<br>urer | Medium       | Fail               | Fail               |
|                   |                   | High         | Fail               | Fail               |

Table 14. Fuel Resistance Data (Tile Test), Source #3

| Additive   | Film Thickness    | Sand Loading  | Humidity<br>13-20% | Humidity<br>40-80% |
|------------|-------------------|---|--------------------|--------------------|
|            |                   | Low   | Pass               | Pass               |
|            | 1/16"             | Medium  | Fail               | Fail               |
|            |                   | High  | Fail               | Fail               |
|            |                   | Low   | Pass               | Pass               |
| Mix Design | 1/8"              | Medium  | Pass               | Pass               |
|            |                   | High  | Fail               | Fail               |
|            |                   | Low   | Pass               | Pass               |
|            | Manufact-<br>urer | Medium  | Pass               | Pass               |
|            |                   | High Fai Low Pas Medium Pas High Fai Low Fai Medium Fai | Fail               | Fail               |
|            |                   | Low   | Fail               | Fail               |
|            | 1/16"             | Medium  | Fail               | Fail               |
|            |                   | High  | Fail               | Fail               |
| Manufact-  |                   | Low   | Pass               | Pass               |
| urer       | 1/8"              | Medium  | Fail               | Pass               |
|            |                   | High  | Fail               | Fail               |
|            |                   | Low   | Pass               | Pass               |
|            | Manufact-<br>urer | Medium  | Fail               | Pass               |
|            | 44                | High  | Fail               | Fail               |

Table 15. Fuel Resistance Data (Tile Test), Source # 4

| Additive   | Film Thickness    | Sand Loading | Humidity<br>13-20% | Humidity<br>40-80% |
|------------|-------------------|--------------|--------------------|--------------------|
|            |                   | Low          | X                  | х                  |
|            | 1/16"             | Medium       | X                  | X                  |
|            |                   | High         | X                  | Х                  |
|            |                   | Low          | x                  | Х                  |
| Mix Design | 1/8"              | Medium       | х                  | Х                  |
|            |                   | High         | x                  | х                  |
|            |                   | Low          | X                  | х                  |
|            | Manufact-<br>urer | Medium       | Х                  | х                  |
|            |                   | High         | х                  | Х                  |
|            |                   | Low          | Fail               | Fail               |
|            | 1/16"             | Medium       | Fail               | Fail               |
|            |                   | High         | Fail               | Fail               |
|            |                   | Low          | Fail               | Pass               |
| Manufact-  | 1/8"              | Medium       | Fail               | Fail               |
| urer       |                   | High         | Fail               | Fail               |
|            |                   | Low          | х                  | х                  |
|            | Manufact-<br>urer | Medium       | х                  | х                  |
|            |                   | Hìgh         | X                  | X                  |

#### Effect of Humidity on Fuel Resistance

In order to evaluate the effect of humidity level on the test results, the data under low and high humidity had to be cross-checked at each level. The data in Tables 12 through 15 show that the effect of humidity level is significant in six out of 54 possible cases. Three cases are in source 1 data (Table 12), two cases are in source 3 data (Table 14), and one case is in source 4 data (table 15). It can be concluded that the effect of humidity level on the results of the fuel resistance test is insignificant.

#### Effect of Sand Loading on Fuel Resistance

The effect of sand loading was evaluated by comparing the results of the fuel resistance test for all three levels of sand loading under each level of humidity and film thickness for both mix designs. The data in Tables 12 through 15 show that the level of sand loading resulted in a change in the result of the test in 29 out of 40 possible cases. All of the mixtures failed the fuel resistance test when high sand loading was used, 58 percent of the mixtures failed when medium sand loading was used; and only 28 percent of the mixtures failed with low sand loading. Moreover 90 percent of the failed cases are at the 1/16 inch film thickness level. It can be concluded that the effect of sand loading on the results of the fuel resistance test is highly significant.

#### CONCLUSIONS

Based on the data collected in these experiments, the following conclusions can be drawn:

- The results of the scuff resistance test are not significantly influenced by the type of substrate. Therefore, an aluminum plate or any available shingle can be used to conduct the scuff resistance test.
- The humidity level has a significant effect on the results of the scuff resistance test. Therefore, the scuff resistance of the coal tar emulsion should be tested under a humidity level which is representative of the environment where the material will be used.
- The results of the peel test are not influenced by the level of humidity under which the material is cured. Therefore a standard humidity level can be set for the peel test.
- The effect of substrate on the results of the freeze-thaw test is insignificant. Therefore, an aluminum plate or any available shingle can be used to conduct the freeze-thaw test.
- The effect of humidity level on the results of the freeze-thaw test is insignificant. Therefore, a standard humidity level can be set for the freeze-thaw test.
- The film thickness is a critical variable in the fuel resistance test. A final film thickness below 1/8 inch may result in the failure of the material. However, multiple applications of the 1/16 inch film thickness provide the same data as a single 1/8 inch film thickness.
- The effect of humidity level on the results of the fuel

resistance test is insignificant. Therefore a standard humidity level can be set for the fuel resistance test.

• The effect of sand loading on the results of the fuel resistance test is highly significant. The data showed that sand loading is a very critical variable and must be controlled very closely.

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## APPENDIX A

## **MIX DESIGN DATA**

## Selection of The Laboratory Mix Design for Source # 1

Note: X indicates that the mixture failed to meet the specifications.

Total Liquids: Check mix for incompatibility between coal tar emulsion and additive Viscosity Test, Limits: 10 - 90 poises.

|        | ADDITIVE |        |      |  |  |
|--------|----------|--------|------|--|--|
| WATER  | Low      | Medium | High |  |  |
| Low    | 57.60    | 16.50  | X    |  |  |
| Medium | 25.40    | X      | X    |  |  |
| High   | 24.00    | X      | X    |  |  |

Composite Mix: Check Workability of Mix, Viscosity Test, Limits: 10 - 90 poises.

|        | ADDITIVE |        |      |  |  |  |
|--------|----------|--------|------|--|--|--|
| WATER  | Low      | Medium | High |  |  |  |
| Low    | 67.10    | 86.50  | X    |  |  |  |
| Medium | 27.80    | X      | x    |  |  |  |
| High   | 21.30    | х      | X    |  |  |  |

Composite Mix: Check Initial and Final Scuff Resistance Scuff Resistance Test, Limits: 100 inch-pounds @ 8 hrs and higher torque @ 24 hrs.

|        |              |     | TIVE |      |   |    |  |
|--------|--------------|-----|------|------|---|----|--|
|        | L            | )W  | Med  | dium |   | gh |  |
| WATER  | Curing Hours |     |      |      |   |    |  |
|        | 8            | 24  | 8    | 24   | 8 | 24 |  |
| Low    | 130          | 135 | 130  | 130  | X | х  |  |
| Medium | 130          | 155 | X    | X    | Х | х  |  |
| High   | 160          | 225 | Х    | Х    | Х | Х  |  |

Composite Mix: Check for Adhesion Between Mix and Substrate Peel Test, Limits: rating of 5A

|        | ADDITIVE |        |      |  |  |
|--------|----------|--------|------|--|--|
| WATER  | Low      | Medium | High |  |  |
| Low    | 5A       | 5A     | X    |  |  |
| Medium | 5A       | X      | Х    |  |  |
| High   | 5A       | X      | x    |  |  |

Composite Mix: Check for resistance to Freeze-Thaw Cracking
Freeze-Thaw Test, Limits: rating of 1 @ 5 cycles & rating of 3 or less @ 10 cycles

|        | ADDITIVE |    |        |      |   |     |
|--------|----------|----|--------|------|---|-----|
|        | La       | )W | Med    | lium | H | igh |
| WATER  |          |    | Cycles |      |   |     |
|        | 5        | 10 | 5      | 10   | 5 | 10  |
| Low    | 1        | 1  | 1      | 1    | х | х   |
| Medium | 1        | 1  | X      | X    | х | х   |
| High   | 0        | 1  | X      | Х    | х | х   |

Composite Mix: Check for Fuel Resistance

Fuel Resistance Test, Limits: Fuel Penetration; Yes or No

|        | ADDITIVE |        |      |  |  |
|--------|----------|--------|------|--|--|
| WATER  | Low      | Medium | High |  |  |
| Low    | Pass     | Pass   | Х    |  |  |
| Medium | Pass     | X      | Х    |  |  |
| High   | Pass     | Х      | Х    |  |  |

### Selection of The Laboratory Mix Design for Source # 2

Total Liquids: Check mix for incompatibility between coal tar emulsion and additive Viscosity Test, Limits: 10 - 90 poises.

|        | ADDITIVE |        |       |  |  |
|--------|----------|--------|-------|--|--|
| WATER  | Low      | Medium | High  |  |  |
| Low    | 22.80    | 49.80  | 35.20 |  |  |
| Medium | х        | 16.90  | 17.90 |  |  |
| High   | X        | 14.50  | 20.80 |  |  |

Composite Mix: Check Workability of Mix,

Viscosity Test, Limits: 10 - 90 poises.

|        | ADDITIVE |        |       |  |  |
|--------|----------|--------|-------|--|--|
| WATER  | Low      | Medium | High  |  |  |
| Low    | 35.60    | 63.20  | 63.60 |  |  |
| Medium | X        | 20.40  | 27.20 |  |  |
| High   | X        | 13.60  | 19.20 |  |  |

Composite Mix: Check Initial and Final Scuff Resistance Scuff Resistance Test, Limits: 100 inch-pounds @ 8 hrs and higher torque @ 24 hrs.

|        |              | ADDITIVE |     |      |     |     |
|--------|--------------|----------|-----|------|-----|-----|
|        | L            | )W       | Мес | lium | H   | igh |
| WATER  | Curing Hours |          |     |      |     |     |
|        | 8            | 24       | 8   | 24   | 8   | 24  |
| Low    | 110          | 200      | 165 | 220  | 190 | 220 |
| Medium | Х            | х        | 130 | 180  | 140 | 175 |
| High   | X            | Х        | 75  | X    | 90  | X   |

Composite Mix: Check for Adhesion Between Mix and Substrate Peel Test, Limits: rating of 5A

|        | ADDITIVE |        |      |  |  |
|--------|----------|--------|------|--|--|
| WATER  | Low      | Medium | High |  |  |
| Lcw    | 5A       | 5A     | 4A   |  |  |
| Medium | X        | 4A     | 4A   |  |  |
| High   | X        | X      | X    |  |  |

Composite Mix: Check for resistance to Freeze-Thaw Cracking
Freeze-Thaw Test, Limits: rating of 1 @ 5 cycles & rating of 3 or less @ 10 cycles

|        |              | ADDITIVE |    |      |   |     |
|--------|--------------|----------|----|------|---|-----|
|        | L            | ow       | Ме | dium | H | igh |
| WATER  | No of Cycles |          |    |      |   |     |
|        | 5            | 10       | 5  | 10   | 5 | 10  |
| Low    | 0            | 1        | 0  | 1    | х | х   |
| Medium | X            | x        | X  | X    | X | х   |
| High   | x            | х        | x  | X    | Х | X   |

Composite Mix: Check for Fuel Resistance

Fuel Resistance Test, Limits: Fuel Penetration; Yes or No

|        | ADDITIVE |        |      |  |  |
|--------|----------|--------|------|--|--|
| WATER  | Low      | Medium | High |  |  |
| Low    | Pass     | Pass   | x    |  |  |
| Medium | x        | x      | x    |  |  |
| High   | x        | х      | x    |  |  |

## Selection of The Laboratory Mix Design for Source # 3

Total Liquids: Check mix for incompatibility between coal tar emulsion and additive Viscosity Test, Limits: 10 - 90 poises.

|        | ADDITIVE |        |       |  |  |
|--------|----------|--------|-------|--|--|
| WATER  | Low      | Medium | High  |  |  |
| Low    | 24.40    | 31.20  | 34.00 |  |  |
| Medium | х        | 10.40  | 11.60 |  |  |
| High   | X        | X      | X     |  |  |

Composite Mix: Check Workability of Mix,

Viscosity Test, Limits: 10 - 90 poises.

|        | ADDITIVE |        |       |  |  |
|--------|----------|--------|-------|--|--|
| WATER  | Low      | Medium | High  |  |  |
| Low    | 29.20    | 35.00  | 35.80 |  |  |
| Medium | X        | 9.70   | 12.00 |  |  |
| High   | X        | x      | Х     |  |  |

Composite Mix: Check Initial and Final Scuff Resistance
Scuff Resistance Test, Limits: 100 inch-pounds @ 8 hrs and higher torque @ 24 hrs.

|        | ADDITIVE     |     |     |        |     |     |  |
|--------|--------------|-----|-----|--------|-----|-----|--|
|        | Low          |     |     | Medium |     | gh  |  |
| WATER  | Curing Hours |     |     |        |     |     |  |
|        | 8            | 24  | 8   | 24     | 8   | 24  |  |
| Low    | 130          | 165 | 100 | 130    | 110 | 155 |  |
| Medium | Х            | X   | 130 | 140    | 120 | 145 |  |
| High   | X            | X   | х   | х      | х   | х   |  |

Composite Mix: Check for Adhesion Between Mix and Substrate Peel Test, Limits: rating of 5A

|        | ADDITIVE |        |      |  |  |
|--------|----------|--------|------|--|--|
| WATER  | Low      | Medium | High |  |  |
| Low    | 5A       | 5A     | 5A   |  |  |
| Medium | X        | 5A     | 5A   |  |  |
| High   | x        | X      | X    |  |  |

Composite Mix: Check for resistance to Freeze-Thaw Cracking
Freeze-Thaw Test, Limits: rating of 1 @ 5 cycles & rating of 3 or less @ 10 cycles

|        | ADDITIVE     |    |        |    |      |    |  |
|--------|--------------|----|--------|----|------|----|--|
|        | Low          |    | Medium |    | High |    |  |
| WATER  | No of Cycles |    |        |    |      |    |  |
|        | 5            | 10 | 5      | 10 | 5    | 10 |  |
| Low    | 0            | 2  | 0      | 1  | 0    | 0  |  |
| Medium | Х            | Х  | 0      | 1  | 0    | 1  |  |
| High   | X            | X  | х      | X  | х    | X  |  |

Composite Mix: Check for Fuel Resistance

Fuel Resistance Test, Limits: Fuel Penetration; Yes or No

|        | ADDITIVE |        |      |  |  |  |
|--------|----------|--------|------|--|--|--|
| WATER  | Low      | Medium | High |  |  |  |
| Low    | Fail     | Pass   | Pass |  |  |  |
| Medium | X        | Fail   | Pass |  |  |  |
| High   | X        | x      | X    |  |  |  |